



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 :  A61K 7/00, 7/50, C11D 17/00, 1/94		A1	(11) International Publication Number: <b>WO 00/59454</b>  (43) International Publication Date: 12 October 2000 (12.10.00)
(21) International Application Number: PCT/EP00/02757  (22) International Filing Date: 28 March 2000 (28.03.00)		(74) Agent: ELLIOTT, Peter, William; Unilever PLC, Patent Department, Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB).	
(30) Priority Data: 09/286,042 5 April 1999 (05.04.99) US		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).	
(71) Applicant ( <i>for AE AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG ZA ZW only</i> ): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).  (71) Applicant ( <i>for all designated States except AE AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG ZA ZW</i> ): UNILEVER NV [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).  (71) Applicant ( <i>for IN only</i> ): HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, 400 020 Mumbai (IN).  (72) Inventors: PUVVADA, Sudhakar; Unilever Home & Personal Care USA, 40 Merritt Boulevard, Trumbull, CT 06611 (US). MITRA, Shuman; 5608 Croydon Avenue, San Jose, CA 95118 (US).		<p><b>Published</b>  <i>With international search report.</i>  <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i> </p>	

(54) Title: LIQUID COMPOSITION WITH ENHANCED LOW TEMPERATURE STABILITY

## (57) Abstract

The invention relates to liquid cleansing compositions in lamellar phase. The use of specific anionic surfactant has been found to enhance both the initial viscosity and the freeze thaw (low temperature) viscosity/stability of the compositions.

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

- 1 -

LIQUID COMPOSITION WITH ENHANCED LOW TEMPERATURE STABILITY

BACKGROUND

5

FIELD OF THE INVENTION

The present invention relates to liquid cleansing compositions of the type typically used in skin cleansing or shower gel compositions which compositions are "structured".  
10 lamellar phase compositions. Such lamellar compositions are characterized by high zero shear viscosity (good for suspending and/or structuring) while simultaneously being very shear thinning such that they readily dispense on pouring. Such compositions possess a "heaping", lotion-like  
15 appearance that conveys signals of enhanced moisturization.

BACKGROUND OF THE INVENTION

The rheological behaviour of all surfactant solutions,  
20 including liquid cleansing solutions, is strongly dependent on the microstructure of the solution, i.e., the shape and concentration of micelles or other self-assembled structures in solution.

25 When there is sufficient surfactant to form micelles, (concentrations above the critical micelle concentration or CMC) spherical, cylindrical (rod-like) or discoidal micelles may, for example, form. As surfactant concentration increases, ordered liquid crystalline phases such as a  
30 lamellar phase, hexagonal phase or cubic phase may form. The lamellar phase, for example, consists of alternating

- 2 -

surfactant bilayers and water layers. These layers are not generally flat but fold to form submicron spherical onion like structures called vesicles or liposomes. The hexagonal phase, on the other hand, consists of long cylindrical 5 micelles arranged in a hexagonal lattice. In general, the microstructures of most personal care products consist of spherical micelles; rod micelles; or a lamellar dispersion.

As noted above, micelles may be spherical or rod-like. 10 Formulations having spherical micelles tend to have a low viscosity and exhibit Newtonian shear behaviour (i.e., viscosity stays constant as a function of shear rate; thus, if easy pouring of product is desired, the solution is less viscous and, as a consequence, it doesn't suspend as well). 15 In these systems, the viscosity increases linearly with surfactant concentration.

Rod micellar solutions tend to be more viscous because movement of the longer micelles is restricted. At a 20 critical shear rate, the micelles align and the solution becomes shear thinning. Addition of salts increases the size of the rod micelles thereby increasing the zero shear viscosity (i.e., the viscosity of the solution when stored in a bottle) which helps suspend particles but also 25 increases the critical shear rate (the point at which the product becomes shear thinning; higher critical shear rates mean that the product is more difficult to pour).

Lamellar dispersions differ from both spherical and 30 rod-like micelles because they can have a high zero shear viscosity (because of the close packed arrangement of

- 3 -

constituent lamellar droplets), yet these solutions are very shear thinning (readily dispense on pouring). That is, the solutions can become thinner than rod micellar solutions at moderate shear rates.

5

In formulating liquid cleansing compositions, therefore, there is the choice of using rod-micellar solutions (whose zero shear viscosity, e.g., suspending ability, is not very good and/or are not very shear thinning); or lamellar dispersions (which have a higher zero shear viscosity, e.g. better suspending, and yet are very shear thinning).

10

To form such lamellar compositions, however, some compromises have to be made. Firstly, generally higher amounts of surfactant are required to form the lamellar phase. Thus, it is often necessary to add auxiliary surfactants and/or salts that are neither desirable nor needed. Secondly, only certain surfactants will form this 20 phase and, therefore, the choice of surfactants is restricted.

15

In short, lamellar compositions are generally more desirable (especially for suspending emollient and for 25 providing consumer aesthetics). However, the use of lamellar compositions is more expensive because they generally require more surfactant and are more restricted in the range of surfactants can be used.

30 When rod-micellar solutions are used, they also often require the use of external structurants to enhance

- 4 -

viscosity and to suspend particles (again, because they have a lower zero shear viscosity than lamellar phase solutions).

For this, carbomers and clays are often used. At higher

shear rates (as in product dispensing, application of

5 product to body, or rubbing with hands), since the rod-micellar solutions are less shear thinning, the viscosity of the solution stays high and the product can be stringy and thick. Lamellar dispersion based products, having higher zero shear viscosity, can more readily suspend emollients

10 and are typically more creamy. Again, however, they are generally more expensive to make (e.g., the surfactants which can be used are more restricted and often higher concentrations of surfactants are required).

15 In general, lamellar phase compositions are easy to identify by their characteristic focal conic shape and oily streak texture while hexagonal phase compositions exhibit an angular fan-like texture. In contrast, micellar phases are optically isotropic.

20

It should be understood that lamellar phases may be formed in a wide variety of surfactant systems using a wide variety of lamellar phase "inducers" as described, for example, in the present applicants' publication, WO  
25 97/05857. Generally, the transition from micelle to lamellar phase is a function of the effective average area of the headgroup of the surfactant, the length of the extended tail, and the volume of tail. Branched surfactants or surfactants with smaller headgroups or bulky tails can be  
30 used as effective ways of inducing transitions from rod micellar to lamellar.

- 5 -

Ways of characterizing lamellar dispersions include measuring the viscosity at low shear rate (using for example a Stress Rheometer) when an additional inducer (e.g., oleic acid or isostearic acid) is used. With higher amounts of inducer, the low shear viscosity will significantly increase.

Another way of measuring lamellar dispersions is using freeze fracture electron microscopy. Micrographs will generally show lamellar microstructure and close packed organization of the lamellar droplets (generally in a size range of about 2 microns).

One problem with certain lamellar phase compositions is that they tend to lose their lamellar stability in colder temperatures (e.g., -18 to 7°C(0 to 45°F)). While not wishing to be bound by theory, this may be because, in cold conditions, the oil droplets become less flexible and the spherical structure characterizing the lamellar interaction breaks giving lamellar sheets instead.

As described in the present applicants' U.S. Serial No. 08/993,497 to Villla, it has been found that the use of certain polymeric emulsifiers (e.g., dipolyhydroxystearate) helps enhance low temperature viscosity.

#### BRIEF DESCRIPTION OF THE INVENTION

Unexpectedly, the present applicants have found that specific anionic surfactants provide enhanced freeze thaw

- 6 -

stability in structured liquid compositions relative to compositions not comprising a branched C<sub>10</sub>-C<sub>22</sub> alkyl, alkali metal ether sulfate. Suitable anionic surfactants include branched C<sub>10</sub>-C<sub>22</sub> alkyl, preferably branched C<sub>10</sub>-C<sub>16</sub> alkyl,

5 alkali metal ether sulfates (i.e., those having at least one branch from the alkyl portion of the alkyl ether sulfate). The alkyl ether sulfate may be used as the sole anionic surfactant or in a mixture of anionic surfactants wherein the branched ether sulfate represents from about 50% to

10 100%, preferably from 51% to 100% of the anionic surfactant.

More specifically, the invention provides a liquid cleansing composition, wherein the liquid is in a lamellar phase, comprising:

15

(a) from 5% to 50% by wt. of a surfactant system comprising:

20

(i) from 0.5 to 25%, preferably from 1 to 15% by wt. of the total composition of one or more anionic surfactants, where the anionic surfactant or at least one of the anionic surfactants comprises a branched C<sub>10</sub>-C<sub>22</sub> alkyl, alkali metal, ether sulfate (where a mixture is used, the branched ether sulfate represents at least about 50% of anionic mixture);

25

(ii) preferably an amphoteric and/or zwitterionic surfactant (e.g., betaine or alkali metal C<sub>8</sub>-C<sub>20</sub> amphoacetate) or a mixture thereof (e.g.,

- 7 -

an amphoteric surfactant or a zwitterionic surfactant or a mixture of an amphoteric surfactant and a zwitterionic surfactant) in an amount of from 0 to 25% by wt., preferably from 0.1 to 20% by wt.; and

(b) from 1 to 15% by wt., preferably from 2% to 10% by wt. of a fatty acid or ester thereof (e.g., a straight chained fatty acid such as lauric acid or a branched fatty acid such as isostearic acid);

wherein said composition has an initial viscosity of greater than 20,000, for example from 20,000 to 300,000 centipoises (cps) measured at 0.5 RPM using T-bar spindle A, preferably from 40,000 cps to 250,000 cps, more preferably from about 50,000 to about 200,000 cps, and a freeze thaw viscosity (measured after at least one cycle, preferably at least two cycles, most preferably three cycles of from -18°C(0°F) to room temperature freeze thaw cycles) defined either by having a viscosity greater than about 30,000 cps, preferably greater than 35,000 (again measured at 0.5 RPM using T-bar spindle A) or by having a percent drop in viscosity relative to initial viscosity of no more than 40%.

Ideally, there should be no change in viscosity from the initial viscosity although this of course is not always possible. The invention may also be defined in this regard, as noted, in that the drop in viscosity after freeze/thaw should be 40% or less, preferably 35% or less than the initial viscosity.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to liquid lamellar  
5 cleansing compositions, particularly liquid cleansing  
compositions comprising:

- (a) from 5% to 50% by wt. of a surfactant system  
comprising one or more anionic surfactants wherein  
10 at least one of the anionic surfactants is a  
branched C<sub>10</sub>-C<sub>22</sub>, preferably C<sub>10</sub>-C<sub>16</sub> alkyl, alkali  
metal ether sulfate and preferably further  
comprising an amphoteric and/or zwitterionic  
surfactant or a mixture thereof; and  
15 (b) from 1% to 15% by wt., preferably from 2 to 10% by  
wt. of a fatty acid or ester thereof (as lamellar  
phase inducing structurant);
- wherein said compositions have an initial  
20 viscosity of greater than 20,000, for example from  
20,000 to 300,000 cps measured at 0.5 RPM using T-bar  
spindle A, preferably from 40,000 cps to 250,000 cps,  
more preferably from about 50,000 to about 200,000 cps,  
and a freeze thaw viscosity (measured after at least  
25 one cycle, preferably at least two cycles, most  
preferably three cycles of -18°C (0°F) to room  
temperature freeze thaw cycles) defined either by  
having a viscosity greater than about 30,000 cps,  
preferably greater than 35,000 (again measured at 0.5  
RPM using T-bar spindle A) or by having a percent drop  
30

- 9 -

in viscosity relative to initial viscosity of no more than 40%.

5    Surfactants

The surfactant system represents from 5 to 50% by weight, preferably from 10 to 40% by wt. of the composition of the invention and comprises:

- 10       (a) one or more anionic surfactants wherein the one, if only one is used, or at least one of the anionic surfactants, if a mixture is used, is a branched C<sub>10</sub>-C<sub>22</sub>, preferably C<sub>10</sub>-C<sub>16</sub> alkyl, alkali metal ether sulfate;
- 15       (b) amphoteric and/or zwitterionic surfactants; and
- (c) optional a nonionic surfactant.

As noted above, the anionic surfactant (or one of the anionic surfactants, if a mixture is used) is a branched C<sub>10</sub>-C<sub>22</sub> alkyl, alkali metal ether sulfate. A preferred ether sulfate is branched C<sub>13</sub> (trideceth) sulfate, particularly branched sodium tridecyl ether sulfate. Branching may occur at one or two or more locations in the alkyl backbone.

25       If used alone, the ether sulfate generally represents from 1 to 25% by wt. of the total composition and, if used as one of two or more anionic surfactants, it will generally represent from 1 to 12.5% by wt. of the total composition.

- 10 -

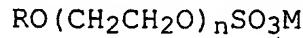
Examples of suitable additional anionic surfactants (which may represent from 0.5% to 12.5% by wt. of the total composition) are set out below.

5 An aliphatic sulfonate, such as a primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) sulfonate, primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) disulfonate, C<sub>8</sub>-C<sub>22</sub> alkene sulfonate, C<sub>8</sub>-C<sub>22</sub> hydroxyalkane sulfonate or alkyl glyceryl ether sulfonate (AGS); or an aromatic sulfonate such as alkyl benzene sulfonate can be used.

10

An alkyl sulfate (e.g., C<sub>12</sub>-C<sub>18</sub> alkyl sulfate) or alkyl ether sulfate (including alkyl glyceryl ether sulfates) can be used. Among suitable alkyl ether sulfates are those having the formula:

15



wherein R is an alkyl or alkenyl having from 8 to 18 carbon atoms, preferably from 12 to 18 carbon atoms, n has an average value of greater than 1.0, preferably from 2 to 20 3; and M is a solubilizing cation such as sodium, potassium, ammonium or substituted ammonium. Ammonium and sodium laurel ether sulfates are preferred.

25 These differ from ether sulfates which are essential to the invention in that they are not branched.

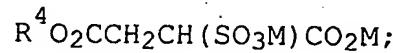
The anionic surfactant may also be an alkyl sulfosuccinate (including mono- and dialkyl, e.g., C<sub>6</sub>-C<sub>22</sub>

- 11 -

sulfosuccinates); an alkyl or acyl taurate, an alkyl or acyl sarcosinate, a sulfoacetate, a C<sub>8</sub>-C<sub>22</sub> alkyl phosphate or phosphate, an alkyl phosphate ester or an alkoxyalkyl phosphate ester, an acyl lactate, a C<sub>8</sub>-C<sub>22</sub> monoalkyl succinate or maleate, a sulphoacetate, or an acyl isethionate.

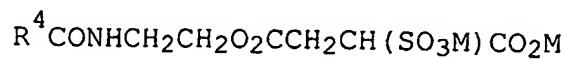
Sulfosuccinates may be monoalkyl sulfosuccinates having the formula:

10



amido-MEA sulfosuccinates of the formula:

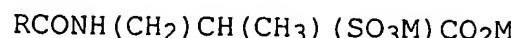
15



wherein R<sup>4</sup> is C<sub>8</sub>-C<sub>22</sub> alkyl and M is a solubilizing cation;

20

amido-MIPA sulfosuccinates of formula:

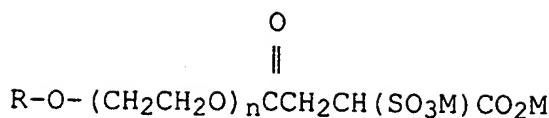


where M is as defined above.

25

Also included are the alkoxylated citrate sulfosuccinates; and alkoxylated sulfosuccinates such as the following:

- 12 -

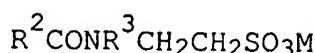


5

wherein n = 1 to 20; and M is as defined above.

Sarcosinates are generally indicated by the formula  
RCON(CH<sub>3</sub>)CH<sub>2</sub>CO<sub>2</sub>M, wherein R is a C<sub>8</sub> to C<sub>20</sub> alkyl and M is a  
10 solubilizing cation.

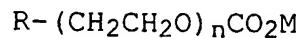
Taurates are generally identified by formula:



15 wherein R<sup>2</sup> is a C<sub>8</sub>-C<sub>20</sub> alkyl, R<sup>3</sup> is a C<sub>1</sub>-C<sub>4</sub> alkyl and M is a solubilizing cation.

Another class of anionic surfactant is carboxylates such as follows:

20



wherein R is C<sub>8</sub> to C<sub>20</sub> alkyl; n is 0 to 20; and M is as defined above.

25

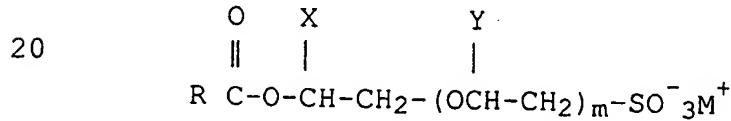
Other carboxylates which can be used include amido alkyl polypeptide carboxylates such as, for example, Monteine LCQ<sup>(R)</sup> by Seppic.

- 13 -

Other surfactants which may be used are the C<sub>8</sub>-C<sub>18</sub> acyl isethionates. These esters are prepared by reaction between an alkali metal isethionate and mixed aliphatic fatty acids having from 6 to 18 carbon atoms and an iodine value of less than 20. At least 75% of the mixed fatty acids have from 12 to 18 carbon atoms and up to 25% have from 6 to 10 carbon atoms.

Acyl isethionates, when present, will generally be present in an amount of from about 0.5-15% by weight of the total composition. Preferably, this component is present in an amount of from about 1 to about 10%.

The acyl isethionate may be an alkoxylated isethionate such as is described in Ilardi et al., U.S. Patent No. 5,393,466, hereby incorporated by reference into the subject application. This compound has the general formula:



wherein R is an alkyl group having from 8 to 18 carbons, m is an integer of from 1 to 4, X and Y are each independently hydrogen or an alkyl group having from 1 to 4 carbons and M<sup>+</sup> is a monovalent cation such as, for example, sodium, potassium or ammonium.

In general the "additional" anionic component will represent from about 1 to 20% by weight of the composition,

- 14 -

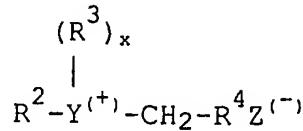
preferably from 2 to 15%, most preferably from 5 to 12% by weight of the composition.

Zwitterionic and Amphoteric Surfactants

5

Zwitterionic surfactants are exemplified by those which can be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds, in which the aliphatic radicals can be a straight or 10 branched chain, and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic group, e.g., carboxy, sulfonate, sulfate, phosphate, or phosphonate. A general formula for these compounds is:

15



20 wherein  $R^2$  contains an alkyl, alkenyl, or hydroxy alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to about 1 glyceryl moiety; Y is selected from the group consisting of nitrogen, phosphorus, and sulfur atoms;  $R^3$  is an alkyl or 25 monohydroxyalkyl group containing from about 1 to about 3 carbon atoms; X is 1 when Y is a sulfur atom, and 2 when Y is a nitrogen or phosphorus atom;  $R^4$  is an alkylene or hydroxyalkylene of from about 1 to about 4 carbon atoms and Z is a radical selected from the group consisting of

- 15 -

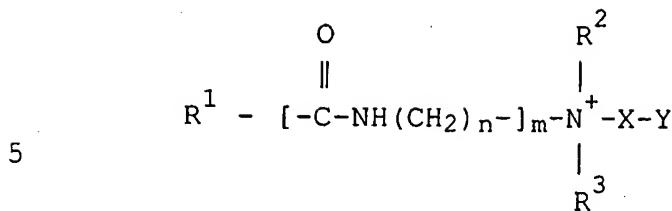
carboxylate, sulfonate, sulfate, phosphonate, and phosphate groups.

Examples of such surfactants include:

- 5       4-[N,N-di(2-hydroxyethyl)-N-octadecylammonio]-butane-1-carboxylate;
- 10      5-[S-3-hydroxypropyl-S-hexadecylsulfonio]-3-hydroxypentane-1-sulfate;
- 15      3-[P,P-diethyl-P-3,6,9-trioxatetradexocylphosphonio]-2-hydroxypropane-1-phosphonate;
- 20      3-[N,N-dipropyl-N-3-dodecoxy-2-hydroxypropylammonio]-propane-1-phosphonate;
- 25      3-(N,N-dimethyl-N-hexadecylammonio)propane-1-sulfonate;
- 30      3-(N,N-dimethyl-N-hexadecylammonio)-2-hydroxypropane-1-sulfonate;
- 35      4-[N,N-di(2-hydroxyethyl)-N-(2-hydroxydodecyl)ammonio]-butane-1-carboxylate;
- 40      3-[S-ethyl-S-(3-dodecoxy-2-hydroxypropyl)sulfonio]-propane-1-phosphonate;
- 45      3-[P,P-dimethyl-P-dodecylphosphonio]-propane-1-phosphonate; and
- 50      5-[N,N-di(3-hydroxypropyl)-N-hexadecylammonio]-2-hydroxy-pentane-1-sulfate.

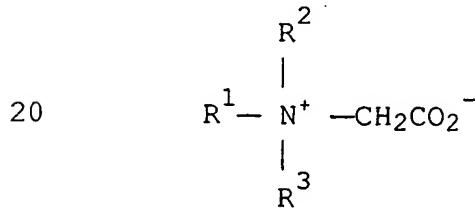
- 25      Amphoteric detergents which may be used in this invention contain at least one acid group. This may be a carboxylic or a sulphonic acid group. They include quaternary nitrogen and therefore are quaternary amido acids. They should generally include an alkyl or alkenyl group of from 7 to 18 carbon atoms. They will usually comply with an overall structural formula:

- 16 -

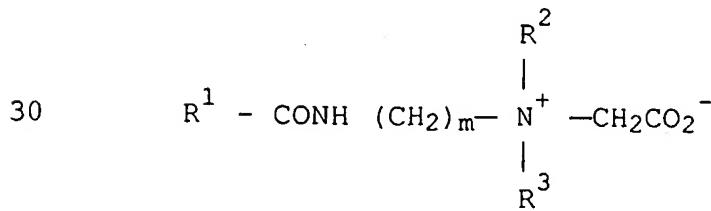


where  $\text{R}^1$  is alkyl or alkenyl having from 7 to 18 carbon atoms;  $\text{R}^2$  and  $\text{R}^3$  are each independently alkyl, hydroxyalkyl or carboxyalkyl having from 1 to 3 carbon atoms;  $n$  is 2 to 4;  $m$  is 0 to 1;  $X$  is alkylene of from 1 to 3 carbon atoms optionally substituted with hydroxyl, and  $Y$  is  $-\text{CO}_2^-$  or  $-\text{SO}_3^-$ .

15        Suitable amphoteric detergents within the above general formula include simple betaines of formula:



25        and amido betaines of formula:

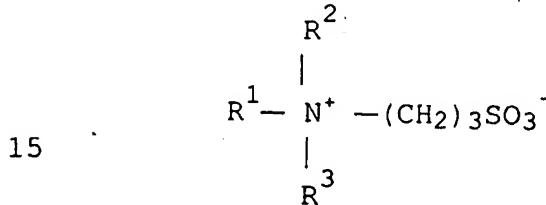


35        where  $m$  is 2 or 3.

- 17 -

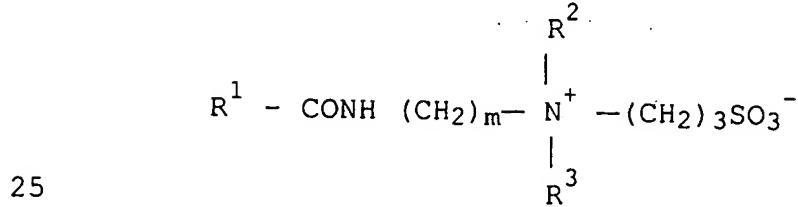
In both formulae R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are as defined previously. R<sup>1</sup> may in particular be a mixture of C<sub>12</sub> and C<sub>14</sub> alkyl groups derived from coconut so that at least half, preferably at least three quarters of the groups R<sup>1</sup> have 10 to 14 carbon atoms. R<sup>2</sup> and R<sup>3</sup> are preferably methyl.

A further possibility is that the amphoteric detergent 10 is a sulphobetaine of formula:



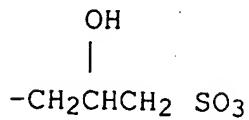
or

20



where m is 2 or 3, or variants of these in which -(CH<sub>2</sub>)<sub>3</sub>SO<sub>3</sub><sup>-</sup> is replaced by

30



- 18 -

In these formulae R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are as defined previously.

The terms amphoacetates and diamphoacetates are also 5 intended to be covered in possible zwitterionic and/or amphoteric compounds which may be used.

The amphoteric/zwitterionic surfactant, when used, generally represents from 0% to 25%, preferably from 0.1 to 10 20% by weight, preferably from 5% to 15% of the composition.

A preferred surfactant system of the invention comprises an unbranched alkyl ether sulfate together with a branched alkyl ether sulfate of the invention, optionally 15 further in combination with betaine and/or amphoacetate.

The surfactant system may also optionally comprise a nonionic surfactant.

20 The nonionic surfactants which may be used includes in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or 25 with propylene oxide. Specific nonionic detergent compounds are alkyl (C<sub>6</sub>-C<sub>22</sub>) phenols-ethylene oxide condensates, the condensation products of aliphatic (C<sub>8</sub>-C<sub>18</sub>) primary or secondary linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the 30 reaction products of propylene oxide and ethylenediamine.

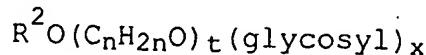
- 19 -

Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

- 5       The nonionic may also be a sugar amide, such as a polysaccharide amide. Specifically, the surfactant may be one of the lactobionamides described in U.S. Patent No. 5,389,279 to Au et al. which is hereby incorporated by reference or it may be one of the sugar amides described in  
10      Patent No. 5,009,814 to Kelkenberg, hereby incorporated into the subject application by reference.

- Other surfactants which may be used are described in U.S. Patent No. 3,723,325 to Parran Jr. and alkyl  
15      polysaccharide nonionic surfactants as disclosed in U.S. Patent No. 4,565,647 to Llenado, both of which are also incorporated into the subject application by reference.

- Preferred alkyl polysaccharides are alkylpolyglycosides  
20      of the formula:



- wherein R<sup>2</sup> is selected from the group consisting of  
25      alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl, and mixtures thereof in which alkyl groups contain from about 10 to about 18, preferably from about 12 to about 14, carbon atoms; n is from 0 to 3, preferably 2; t is from 0 to about 10, preferably 0; and x is from 1.3 to about 10, preferably  
30      from 1.3 to about 2.7. The glycosyl is preferably derived

- 20 -

from glucose. To prepare these compounds, the alcohol or alkylpolyethoxy alcohol is formed first and then reacted with glucose, or a source of glucose, to form the glucoside (attachment at the 1-position). The additional glycosyl 5 units can then be attached between their 1-position and the preceding glycosyl units 2-, 3-, 4- and/or 6-position, preferably predominantly the 2-position.

Nonionic surfactants represent from 0 to 10% by wt. of the composition.

10

Structurant

The compositions of the invention utilize from about 1% to 15% by wt., preferably from 2 to 10% by wt. of a 15 structuring agent which works in the compositions to form a lamellar phase. Such a lamellar phase enables the compositions to suspend particles more readily (e.g., emollient particles) while still maintaining good shear thinning properties. The lamellar phase also provides 20 consumers with desired rheology ("heaping").

The structurant is typically a fatty acid or ester derivative thereof.

25 Examples of fatty acids which may be used are C<sub>10</sub>-C<sub>22</sub> acid (e.g. lauric, oleic etc.), isostearic acid, linoleic acid, linolenic acid, ricinoleic acid, elaidic acid, arachidonic acid, myristoleic acid and palmitoleic acid. Ester derivatives include propylene glycol isostearate, 30 propylene glycol oleate, glyceryl isostearate, glyceryl oleate and polyglyceryl diisostearate.

- 21 -

Oil/Emollient

One of the principle benefits of the invention is the  
5 ability to suspend oil/emoilient particles in a lamellar  
phase composition. The following oil/emoillients may  
optionally be suspended in the compositions of the  
invention.

10 Vegetable oils: Arachis oil, castor oil, cocoa butter,  
coconut oil, corn oil, cotton seed oil, olive oil, palm  
kernel oil, rapeseed oil, safflower seed oil, sesame seed  
oil and soybean oil.

15 Esters: Butyl myristate, cetyl palmitate, decyloleate,  
glyceryl laurate, glyceryl ricinoleate, glyceryl stearate,  
glyceryl isostearate, hexyl laurate, isobutyl palmitate,  
isocetyl stearate, isopropyl isostearate, isopropyl laurate,  
isopropyl linoleate, isopropyl myristate, isopropyl  
20 palmitate, isopropyl stearate, propylene glycol monolaurate,  
propylene glycol ricinoleate, propylene glycol stearate, and  
propylene glycol isostearate.

Animal Fats: acetylated lanolin alcohols, lanolin,  
25 lard, mink oil and tallow.

Other examples of oils/emoillients include mineral oils,  
petrolatum, silicone oils such as dimethyl polysiloxane,  
lauryl and myristyl lactate.

- 22 -

The emollient/oil is generally used in an amount of from about 1 to 20%, preferably from 1 to 15% by wt. of the composition. Generally, it should represent no more than 20% of the composition.

5

In addition, the compositions of the invention may include optional ingredients as follows:

Organic solvents, such as ethanol; auxiliary  
10 thickeners, sequestering agents, such as tetrasodium ethylenediaminetetraacetate (EDTA), EHDP or mixtures in an amount of from 0.01 to 1%, preferably from 0.01 to 0.05%; and coloring agents, opacifiers and pearlizers such as zinc stearate, magnesium stearate, TiO<sub>2</sub>, EGMS (ethylene glycol monostearate) or Lytron 621 (Styrene/Acrylate copolymer);  
15 all of which are useful in enhancing the appearance or cosmetic properties of the product.

The compositions may further comprise antimicrobials  
20 such as 2-hydroxy-4,2'4' trichlorodiphenylether (DP300); preservatives such as dimethyloldimethylhydantoin (Glydant XL1000), parabens, sorbic acid etc.

The compositions may also comprise coconut acyl mono-  
25 or diethanol amides as suds boosters, and strongly ionizing salts such as sodium chloride and sodium sulfate may also be used to advantage.

Antioxidants such as, for example, butylated  
30 hydroxytoluene (BHT) may be used advantageously in amounts of about 0.01% or higher if appropriate.

- 23 -

Cationic conditioners which may be used include Quatrisoft LM-200 Polyquaternium-24, Merquat Plus 3330 - Polyquaternium 39; and Jaguar<sup>(R)</sup> type conditioners.

5

Other optional ingredients which may be added are the deflocculating polymers such as those described in U.S. Patent No. 5,147,576 to Montague, hereby incorporated by reference.

10

Other ingredients which may be included are exfoliants such as polyoxyethylene beads, walnut sheets and apricot seeds.

15

The compositions of the invention, as noted above, are lamellar compositions. In particular, the lamellar phase represents from 30 to 80%, preferably from 40 to 70% of the total phase volume. The phase volume may be measured, for example, by conductivity measurements or other measurements 20 which are well known to those skilled in the art. While not wishing to be bound by theory, higher phase volume is believed to provide better suspension of emollients.

25 The invention will now be described in greater detail by way of the following non-limiting Examples. The Examples are for illustrative purposes only and not intended to limit the invention in any way.

30 Except in the operating and comparative Examples, or where otherwise explicitly indicated, all number in this description indicating amounts or ratios of materials or

- 24 -

conditions or reaction, physical properties of materials and/or use are to be understood as modified by the word "about".

5 Where used in the specification, the term "comprising" is intended to include the presence of stated features, integers, steps, components, but not to preclude the presence or addition of one or more features, integers, steps, components or groups thereof.

10

All percentages in the specification and Examples are intended to be by weight unless stated otherwise.

#### EXAMPLES

15

Tests on lamellar structured shower gel compositions were conducted on the following base compositions:

#### Base

Ingredient	% by Wt.
Sodium Trideceth Sulfate	15%
Sodium Lauryl Ether Sulfate (SLES)	0-10%
Amphoteric Surfactant (e.g., Sodium Lauroamphoacetate)	5-15%
Oil/Emollient (e.g., Sunflower Seed Oil; Silicone; Petrolatum)	0 - 15%
Opacifier/Colorant	0 - 2%
Perfume/Preservative	0 - 3%
Lamellar Inducing Fatty Acid (e.g., Isostearic Acid)	1 - 8%

20

Viscosity measurements were made in accordance with the following protocol:

- 25 -

### Viscosity Measurement

#### Scope:

5 This method covers the measurement of the viscosity of the finished product. It is used to measure the degree of structuring of the product.

#### Apparatus:

10 Brookfield RVT Viscometer with Helipath Accessory;  
Chuck, weight and closer assembly for T-bar attachment;  
T-bar Spindle A;  
Plastic cups diameter greater than 2.5 inches.

15

#### Procedure:

1. Verify that the viscometer and the helipath stand are level by referring to the bubble levels on the back of  
20 the instrument.
2. Connect the chuck/closer/weight assembly to the Viscometer (Note the left-hand coupling threads).
- 25 3. Clean Spindle A with deionized water and pat dry with a Kimwipe sheet. Slide the spindle in the closer and tighten.
4. Set the rotational speed at 0.5 RPM. In case of a  
30 digital viscometer (DV) select the % mode and press autozero with the motor switch on.

- 26 -

5. Place the product in a plastic cup with inner diameter of greater than 2.5 inches. The height of the product in the cup should be at least 3 inches. The temperature of the product should be 25°C.

5

6. Lower the spindle into the product (~1/4 inches). Set the adjustable stops of the helipath stand so that the spindle does not touch the bottom of the plastic cup or come out of the sample.

10

7. Start the viscometer and allow the dial to make one or two revolutions before turning on the Helipath stand. Note the dial reading as the helipath stand passes the middle of its downward traverse.

15

8. Multiply the dial reading by a factor of 4,000 and report the viscosity reading in cps.

EXAMPLES 1-3

20

The following Table clearly shows the effect of sodium trideceth sulfate (STDS) in enhancing F/T stability of a structured liquid formulation:

- 27 -

Example	1	2	3
Sodium tricedeth sulfate	10	0	10
Sodium lauryl ether sulfate	0	10	0
Cocoamidopropyl betaine	0	0	0
Sodium lauro amphoacetate	15	15	15
Sunflower oil	0	0	0
Lauric acid	3.2	3.2	0
Isostearic acid	0	0	6
Citric acid	1.7	1.7	1.7
R/T viscosity (T-bar), cps	57600	64000	236800
F/T viscosity (T-bar), cps	38400	9600	227200
% drop	33	85	4

- Comparing Examples 1 and 2, we find a 33% drop in  
 5 viscosity in the formulations with STDS versus an 85% drop  
 in viscosity in the formulations without STDS. Formulation  
 3 which also uses STDS with a soluble structurant  
 (isostearic acid) undergoes a minimal (4%) decrease in  
 viscosity under F/T conditions.

10

#### EXAMPLES 4-5 (Lower Surfactant Level)

Example	4	5
Sodium tricedeth sulfate	6	0
Sodium lauryl ether sulfate	0	6
Cocoamidopropyl betaine	0	0
Sodium lauro amphoacetate	9	9
Sunflower oil	15	15
Lauric acid	3.2	3.2
Isostearic acid	0	0
Citric acid	1.7	1.7
R/T viscosity (T-bar), cps	294400	48000
F/T viscosity (T-bar), cps	291200	19200
% drop	1	60

Similar trends to those of Examples 1-3 are found in formulations with and without STDS when the total actives are reduced to 15% (compared to 25% active in Examples 1-3). In this case, the differences in F/T viscosities are more dramatic (Examples 4 and 5). For example, Example 4 using STDS undergoes a mere 1% decrease in viscosity whereas Example 5, which doesn't contain STDS, undergoes a 60% decrease in F/T viscosity.

10 EXAMPLES 6-8 (Use of Different Amphotericics)

Example	6	7	8
Sodium tricedeth sulfate	10	0	10
Sodium lauryl ether sulfate	0	10	0
Cocoamidopropyl betaine	15	15	15
Sodium lauro amphoacetate	0	0	0
Sunflower oil	0	0	0
Lauric acid	3.2	3.52	0
Isostearic acid	0	0	5
Citric acid	1.7	1.7	1.7
R/T viscosity (T-bar), cps	25600	22400	64000
F/T viscosity (T-bar), cps	16000	6400	51200
% drop	38	72	20

When betaine was used as the amphoteric surfactant, formulations prepared with STDS also exhibited improved F/T stability. For example, the viscosity drop in Examples 6 (with STDS) and 7 (without STDS) were 38% and 72% respectively. Example 8 (similar to Sample 6) using isostearic acid undergoes a 20% drop in viscosity under F/T conditions.

- 29 -

EXAMPLES 9-10 (Lower Surfactant; Betaine)

Example	9	10
Sodium tricedeth sulfate	6	0
Sodium lauryl ether sulfate	0	6
Cocoamidopropyl betaine	9	9
Sodium lauro amphoacetate	0	0
Sunflower oil	10	10
Lauric acid	3.6	3.6
Isostearic acid	0	0
Citric acid	1.4	1.4
R/T viscosity (T-bar), cps	67200	60800
F/T viscosity (T-bar), cps	48000	16000
% drop	29	74

- 5       The differences in viscosity drop with and without STDS (Examples 9 and 10 respectively) were even more dramatic when the total surfactant levels were reduced to 15%. The amphoteric surfactant was betaine. Example 9 (using STDS) went through a 29% viscosity decrease while the viscosity of  
10      Example 10 (without STDS) decreased by 74%.

- 30 -

EXAMPLES 11-12 (Anionic Mixtures)

Example	11	12
Sodium tricedeth sulfate	4.5	4.5
Sodium lauryl ether sulfate	4.5	4.5
Cocoamidopropyl betaine	0	0
Sodium lauro amphoacetate	13.5	13.5
Sunflower oil	5	5
Lauric acid	3	3.2
Isostearic acid	0	0
Glycerine	2	2
Citric acid	1.9	1.6
Fragrance	1	1
Guar hydroxypropyl trimonium chloride	0.5	0.5
DMDM Hydantoin	0.2	0.2
EDTA	0.02	0.02
EHDP	0.02	0.02
R/T viscosity (T-bar), cps	154000	134000
F/T viscosity (T-bar), cps	151000	126000
% drop	2	6

- Formulations 11 and 12 were prepared with a 1:1  
 5 (active) combination of STDS and SLES as the anionic surfactants, differing in the levels of lamellar structurants. The F/T viscosity drop for both these formulations was between 2-6%.

CLAIMS

1. A liquid lamellar cleansing composition  
5 comprising:
  - (a) from 5% to 50% by wt. of a surfactant system comprising:
    - (i) one or more anionic surfactants; where the one anionic surfactant or at least one of the anionic surfactants is a branched C<sub>10</sub>-C<sub>22</sub> alkyl, alkali metal ether sulfate;
    - (ii) from 0.1 to 25% by wt. of the total composition of an amphoteric surfactant or a zwitterionic surfactant or mixtures thereof;  
15 and
  - (b) from 1% to 15% by wt. of a fatty acid or an ester thereof;
- wherein said composition has an initial viscosity  
20 of from 20,000 to 300,000 cps, measured at 0.5 RPM using T-bar spindle A; and a freeze-thaw viscosity defined either by having a viscosity greater than about 30,000 cps also measured at 0.5 RPM using T-bar spindle A; or by having a percent drop of  
25 viscosity relative to the initial viscosity of no more than about 40%.
2. A composition according to claim 1 comprising at least two anionic surfactants wherein one of the anionic  
30 surfactants is acyl isethionate.

3. A composition according to claim 1 or 2, comprising at least two anionic surfactants wherein about 50% of the anionic surfactant system is said branched alkyl, alkali metal ether sulfate.

5

4. A composition according to any one of the preceding claims, wherein said branched alkyl, alkali metal ether sulfate is a C<sub>10</sub>-C<sub>16</sub> alkyl ether sulfate.

10

5. A composition according to claim 4, wherein said branched C<sub>10</sub>-C<sub>16</sub> alkyl, alkali metal sulfate is an alkali metal trideceth sulfate.

6. A composition according to any one of the preceding claims, comprising from 0.1 to 25% by wt. of the composition of the anionic surfactant or surfactants.

7. A composition according to any one of the preceding claims, wherein the amphoteric surfactant is betaine.

8. A composition according to any one of claims 1 to 6, wherein the amphoteric surfactant is lauro amphoacetate.

25

9. A composition according to any one of the preceding claims, wherein the structurant is isostearic acid.

10. A composition according to any one of the preceding claims, comprising from 2% to 10% by wt. of a fatty acid.

- 33 -

11. A composition according to any one of the preceding claims, wherein the initial viscosity is from 40,000 to 250,000 cps.

5

12. A composition according to claim 11, wherein the initial viscosity is from 50,000 to 200,000 cps.

13. A composition according to any one of the preceding claims, wherein the percentage drop in viscosity between the initial viscosity and the final viscosity is 35% or less.

14. A composition according to any one of the preceding claims, wherein the lamellar phase volume presents from 30 to 80% of total phase volume.

# INTERNATIONAL SEARCH REPORT

Inte onal Application No  
PCT/EP 00/02757

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC 7 A61K7/00 A61K7/50 C11D17/00 C11D1/94

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, A	WO 99 32069 A (UNILEVER) 1 July 1999 (1999-07-01) cited in the application page 7, paragraph 5 -page 15, paragraph 3 page 19, paragraphs 3,4 examples 1-4 claims 1-4,9,10 --- A WO 97 05857 A (UNILEVER) 20 February 1997 (1997-02-20) cited in the application page 6, paragraph 5 -page 13, paragraph 2 page 16, paragraph 1 -page 17, paragraph 1 examples X,XI claims 1,5 ---	1,2,6-13 1,2,6-10 -/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

18 July 2000

Date of mailing of the international search report

31/07/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.  
Fax: (+31-70) 340-3016

Authorized officer

Bertran Nadal, J

# INTERNATIONAL SEARCH REPORT

Inte onal Application No  
PCT/EP 00/02757

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 98 13022 A (UNILEVER) 2 April 1998 (1998-04-02) page 4 page 6, paragraph 5 -page 10, paragraph 2 page 13, last paragraph -page 14, paragraph 2 examples 1-4 ---	1-3,6-13
A	WO 98 11871 A (PROCTER & GAMBLE) 26 March 1998 (1998-03-26) page 3, paragraph 3 -page 4, paragraph 5 examples 1-4 ---	1-3,6-8, 14
A	US 4 075 129 A (FUJINO TAKASHI ET AL) 21 February 1978 (1978-02-21) column 1, line 60 -column 2, line 43 example 5 -----	1,3-6

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte. onal Application No

PCT/EP 00/02757

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
WO 9932069	A	01-07-1999	AU	2271999 A	12-07-1999
WO 9705857	A	20-02-1997	AU	719223 B	04-05-2000
			AU	6737396 A	05-03-1997
			BR	9609893 A	25-05-1999
			CA	2222274 A	20-02-1997
			CN	1192675 A	09-09-1998
			CZ	9800355 A	13-05-1998
			EP	0839023 A	06-05-1998
			HU	9901681 A	29-11-1999
			JP	11513053 T	09-11-1999
			PL	324889 A	22-06-1998
			US	6077816 A	20-06-2000
			US	5952286 A	14-09-1999
WO 9813022	A	02-04-1998	AU	4383997 A	29-05-1998
			CZ	9901042 A	15-09-1999
			EP	0928186 A	14-07-1999
			PL	332377 A	13-09-1999
			US	5962395 A	05-10-1999
			ZA	9708189 A	11-03-1999
WO 9811871	A	26-03-1998	CN	1233949 A	03-11-1999
			CZ	9900970 A	11-08-1999
			JP	2000503026 T	14-03-2000
US 4075129	A	21-02-1978	JP	1253965 C	12-03-1985
			JP	51109002 A	27-09-1976
			JP	54013242 B	29-05-1979
			BE	839339 A	01-07-1976
			BR	7601645 A	21-09-1976
			DE	2610338 A	07-10-1976
			FR	2304666 A	15-10-1976
			GB	1504843 A	22-03-1978
			IT	1057010 B	10-03-1982
			MX	3670 E	21-04-1981